Ethyl Formate As a Postharvest Fumigant for Selected Pests of Table Grapes

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ABSTRACT Ethyl formate (EF) in combination with CO_2 was tested for control of arthropods commonly infesting California table grapes. In addition, table grape tolerance to EF exposure was evaluated. LC_{99} estimates were developed for target pests by using a range of EF concentrations (0.04–4.7% EF). Response to treatments varied greatly between species, as well as life stages within species. Western flower thrips, *Frankliniella occidentalis* (Pergande), and adult and crawler stages of grape mealybug, *Pseudococcus maritimus* (Ehrhorn), were most susceptible to EF treatments. Least susceptible were grape mealybug eggs; Pacific spider mite, *Tetranychus pacificus* McGregor; and omnivorous leafroller, *Platynota stultana* Walsingham. The LC_{99} for target pests fell within the range of EF concentrations tolerated by table grapes with the exception of 1-, 3-, and 5-d-old omnivorous leafroller pupae.

KEY WORDS carbon dioxide, controlled atmosphere, *Frankliniella occidentalis*, *Platynota stultana*, *Pseudococcus maritimus*, quarantine, *Tetranychus pacificus*, 'Thompson Seedless'

Investigations into the use of naturally occurring plant volatiles as potential fumigants for postharvest treatment of arthropods has increased due to restrictions governing the use of methyl bromide as mandated by the Montreal Protocol on substances that deplete the ozone layer (TEAP 2000). Fruits naturally produce volatile compounds that are important for aromatic and flavor characteristics (Nursten 1970). Plant volatiles such as ethyl formate (EF) have been shown to have insecticidal properties (Vincent and Lindgren 1971, Aharoni et al. 1987, Rohitha et al. 1993). One important advantage of using volatiles such as EF for fumigation is that residues found on treated commodities are found only in trace amounts (Muthu et al. 1984, Desmarchelier and Ren 1999). Ethyl formate occurs naturally in a variety of products, including essential oils of grasses, beer, rice, beef, and cheese (Desmarchelier 1999). Volatile components of grapes and wine also include EF (Hiroyasu et al. 1972).

The Food and Drug Administration (FDA 2004) has reviewed the use of EF as a flavoring agent and has characterized this compound as generally recognized as safe. Ethyl formate has been widely used as a fumigant for pests associated with dried fruit (Vincent and Lindgren 1971, 1972; Desmarchelier et al. 1999). Ethyl formate was previously registered in the United States for control of several stored-product pests, including confused flower beetle, *Tribolium confusum* Jacqueline du Val; and raisin moth, *Ephestia figulilella* Gregson. Registrations for the use of EF at both federal and state levels have expired in the United States.

A variety of arthropods infest California table grapes. Although they may be present at or below economic threshold levels in table grapes, many export markets have designated several of these arthropods as actionable pests, demanding quarantine levels of control. Here, we report the effectiveness of various concentrations of EF alone and in combination with elevated carbon dioxide against selected arthropods commonly associated with California table grapes. Western flower thrips, *Frankliniella occidentalis* (Pergande), is a common pest of many agricultural and horticultural crops in California, and both its feeding and oviposition on berries can cause damage. Thrips also can cause foliar damage but rarely to a level requiring chemical control (Flaherty et al. 1992). Grape mealybug, *Pseudococcus maritimus* (Ehrhorn), produces two broods per year and overwinters as eggs or crawlers. In the spring, crawlers move onto expanding green tissue and mature during May and June. Females then return to old wood and lay eggs. The eggs hatch during June and July, and most crawlers move to feed on fruit and foliage. It is the second brood that causes most of the fruit damage. During August and September, females are often found in the fruit and can lay eggs in berry clusters (Flaherty et al. 1992). Pacific spider mite, *Tetranychus pacificus* McGregor, feeds on both the foliage and berries of table grapes and can cause significant damage when found in high numbers (Flaherty et al. 1992). Omniv-

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Table 1. LC_{99} and 95% CI for various arthropod pests exposed to EF for 1 h at 24°C in air or in 10% CO_2 balance air

Target pest	Life stage	Atmosphere	n^a	Slope ± SE	LC_{99} (% EF ± 95% CI)
Western flower thrips	Egg	Air	1,547[b]		1.99 (1.84–2.14)
		10% CO_2	2,360[b]		2.40 (2.09–2.71)
	Second instar	Air	16,559	4.97 ± 0.76	0.60 (0.47–1.0)
		10% CO_2	15,621	4.77 ± 0.70	0.62 (0.48–1.1)
	Prepupae	Air	6,457	4.54 ± 0.68	0.87 (0.59–2.0)
		10% CO_2	20,940	5.29 ± 0.83	0.43 (0.34–0.52)
	Adult	Air	11,773	8.40 ± 0.75	0.33 (0.31–0.37)
		10% CO_2	6,593	5.29 ± 0.62	0.20 (0.16–0.28)
Pacific spider mite	Egg	Air	8,768	6.95 ± 0.50	1.92 (1.78–2.13)
		10% CO_2	9,230	6.51 ± 0.79	1.91 (1.67–2.39)
	Protonymph	Air	8,498	5.38 ± 0.46	2.26 (1.97–2.74)
		10% CO_2	5,636	4.90 ± 0.37	1.95 (1.73–2.28)
	Deutonymph	Air	7,214	5.08 ± 0.49	2.39 (2.02–3.07)
		10% CO_2	4,847	5.10 ± 0.51	2.44 (2.01–3.29)
	Adult	Air	9,670	5.44 ± 0.57	3.73 (3.18–5.88)
		10% CO_2	10,414	5.09 ± 0.51	3.45 (2.99–4.26)
Grape mealybug	Egg	Air	9,862	3.26 ± 0.66	4.85 (3.52–9.28)
		10% CO_2	8,175	4.71 ± 0.50	3.48 (3.16–3.87)
	Crawlers	Air	10,888	5.95 ± 0.75	0.82 (0.68–1.12)
		10% CO_2	10,058	5.79 ± 1.76	0.07 (0.03–0.10)
	Adult	Air	787	9.38 ± 0.76	1.79 (1.69–1.91)
		10% CO_2	723	7.03 ± 0.94	1.29 (1.15–1.39)

[a] Total number treated over three replications.
[b] Actual number counted, unknown number of total subjects.

orous leafroller, *Platynota stultana* Wasingham, is a major vineyard pest in California. It feeds on flowers and developing berries and feeding damage provides entry for secondary rot organisms that further damage clusters (Flaherty et al. 1992).

Western flower thrips, grape mealybug, omnivorous leaf roller, and pacific spider mite are all considered arthropod pests of concern by the Australian Quarantine and Inspection Service (AQIS 2000). Methyl bromide (MeBr) is currently recommended as a mandatory treatment to control all four species. Western flower thrips is considered a low quarantine risk and would be subject only to inspection, however, the remaining three pests are considered high quarantine risk and MeBr treatment would be required. Another treatment option for quarantine security is desired. Our objective was to determine if EF would be effective as a MeBr alternative for table grapes.

Susceptibility to many types of control measures varies greatly with target pest age, therefore a range of life stages for each target pest was tested with EF. Preliminary arthropod tests indicated that LC_{99} concentrations of EF would be in a range well tolerated by table grapes. Additionally, arthropod tests had indicated that longer exposure times might be necessary to achieve complete mortality for omnivorous leafroller and certain other pest life stages.

Materials and Methods

Target Pests. Western flower thrips were reared in the laboratory on fresh green beans, *Phaseolus vulgaris* L., at a constant temperature of 24°C and a photoperiod of 12:12 [L:D] h. For exposure to EF, a section of green bean with 100–200 second instars, prepupae, or adult thrips were placed in a 59-ml plastic portion cup (Solo Cup Company, Urbana, IL) with a vented lid. For the egg stage, adult thrips were placed in a cup with fresh green beans and females were allowed to oviposit in the beans for 24 h. Green beans containing thrips eggs were then cut into 3-cm sections, randomized, and placed (two sections per cup) in portion cups for treatment or experimental control. After treatment, all stages other than the egg stage were held for 48 h at 24°C and then evaluated for mortality. Green beans containing eggs were held for 14 d at 24°C at (50–60% RH) to ensure all viable eggs had eclosed, then they were evaluated for mortality.

Pacific spider mites were reared in the laboratory on cotton, *Gossypium hirsutum* L., seedlings at a constant temperature of 24°C and a photoperiod of 24:0 [L:D] h. For exposure to EF, cotton cotyledons infested with 50–200 protonymph, deutonymph, or adult mites were placed in portion cups with vented lids. For the egg stage, fresh plants were placed adjacent to infested plants and left overnight (18 h). The next day, eggs were gently removed from the cotton leaves with a small paint brush and placed in a vented petri dish. A thin layer of Tangle trap (The Tanglefoot Company, Grand Rapids, MI) was applied around the bottom edge of the dish to prevent hatched mites from escaping. After treatment, all stages other than the egg stage were held for 48 h at 24°C (50–60% RH) and then evaluated for mortality. Petri dishes containing eggs were placed at 24°C (50–60% RH) and held for 14 d at 24°C to ensure all viable eggs had eclosed, then they were evaluated for mortality.

Adult female grape mealybugs, *Pseudococcus maritimus* (Ehrhorn), were field collected from grapevines in the Napa Valley region of California. Mealybugs were either treated as adults (10–20 per portion cup) or held singly in portion cups with no food source until

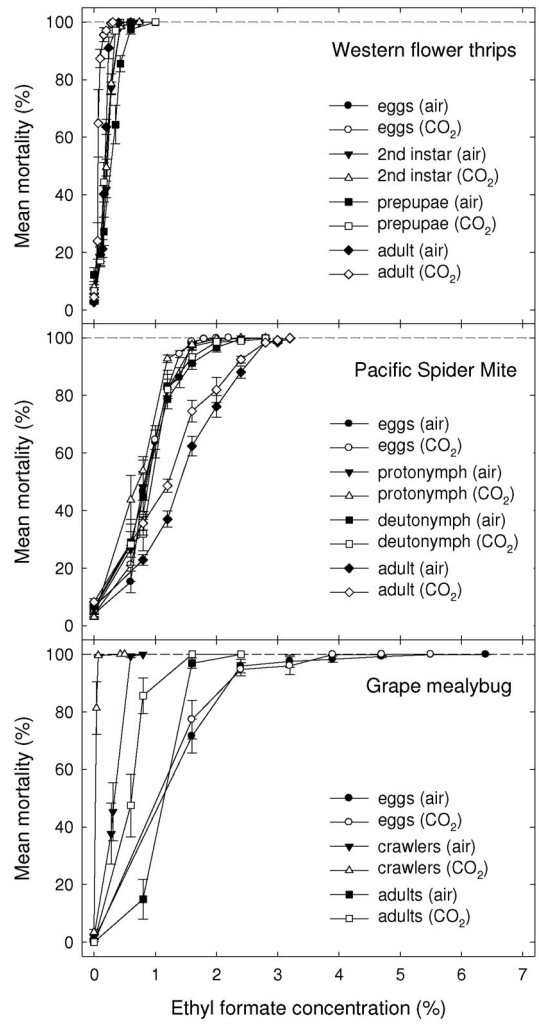

Fig. 1. Mortality of various arthropod pests exposed to a range of EF concentrations for 1 h at 24°C in air or in 10% CO_2 balance air.

the initiation of an ovisac. Females were allowed to lay eggs for 48 h, after which time they were removed from the cup with as little disturbance to the ovisac as possible. The eggs (≈100 per cup) were then held for 5 d before treatment (6–7-d-old eggs). To obtain crawlers, eggs were allowed to hatch and crawlers (≈175 per cup) were separated from unhatched eggs and placed in a vented portion cup for treatment. After treatment, adult and crawler stage mealybugs were held for 48 h at 24°C (50–60% RH) and then evaluated for mortality. Mealybug eggs were held for 3 mo at 24°C (≥90% RH) to ensure all viable eggs had eclosed and then evaluated for mortality.

Omnivorous leafrollers were reared on a modified lima bean diet (Yokoyama et al. 1987) with a photoperiod of 16:8 (L:D) h at 28°C. For exposure to EF, larvae (third or fifth instars) or pupae (1-, 3-, or 5-d-old) were placed in portion cups with vented lids. A small amount of diet was placed in the cup with the third and fifth instars for treatment. For the egg stage, adult females were caged and allowed to lay eggs on wax paper. The wax paper was then removed, cut into small pieces containing an egg mass (≈50 eggs), and then placed in portion cups with vented lids for treatment. Eggs were collected and treated after 1, 2, or 4 d. After exposure to EF, larval stages were held at 24°C for 48 h until evaluation. After treatment, egg and pupal stages were held at 24°C (95–100% RH) in a plastic container (lidded, but not airtight) with damp paper towels. Cups containing eggs or pupae were held for 1 mo or 2 wk, respectively, at 24°C (≥90% RH) to ensure all viable eggs had eclosed, and then they were evaluated for mortality.

For exposure to EF, a replicate consisted of two portion cups or two petri dishes containing target pests placed inside 3.8-liter glass treatment jars sealed with rubber stoppers. For treatments requiring 10% CO_2, jars containing target pests were flushed with 10% CO_2, balance air (>90% RH), then sealed. A partial vacuum was pulled with a syringe, and reagent grade (99.5% purity) liquid EF at 2°C (Fisher, Pittsburgh, PA) was injected through a rubber septum covering an inlet port in the rubber stopper onto filter paper affixed to the underside of the stopper. A separate outlet port in the rubber stopper was used to sample the airspace in the treatment jars for EF concentrations through a 3-mm-diameter plastic tube extending approximately halfway into the jar. Each target pest was exposed to at least six concentrations (three replications per concentration) of EF at 24°C between 0.04 and 4.7%, resulting in a range of mortality between 0 and 100%. All target pests were exposed to EF for 1 h with the exception of omnivorous leafrollers, which were exposed to EF for 2 h. Airspace samples (1 ml) were obtained during tests using a glass, gastight, 1-ml syringe (Hamilton Co., Reno, NV). Ethyl formate concentration was measured using a gas chromatograph (GC-9AM, Shimadzu Scientific Instruments, Columbia, MD) fitted with a 60/80 carbopack column with 5% carbowax (Supelco, Bellefonte, PA), flame ionization detector at 250°C, injection port temperature of 250°C (N_2 as carrier gas), and oven temperature of 85°C.

Lethal concentration estimates were performed using the Probit model developed by Finney (1971). For western flower thrips eggs, the LC_{99} was determined from estimated mortality, which was based on subtracting the number of hatched insects in each treatment from hatched insects in the control. The data were log normalized by transformation, and analysis of covariance was used to calculate the LC_{99} (SAS Institute 2002–2003). Differences between the LC_{99} of different pests and/or life stages were considered significant if the 95% confidence intervals (CI) did not overlap.

Ethyl Formate Treatment of Table Grapes. 'Thompson Seedless' table grapes were obtained from a local grower and sorted for quality. Grapes clusters were randomly assigned to a treatment (or untreated

Table 2. LC_{99} and 95% CI for various life stages of omnivorous leafroller exposed to EF for 2 h at 24°C in air or in 10% CO_2 balance air

Life stage	Atmosphere	n^a	Slope ± SE	LC_{99} (% EF ± 95% CI)
Egg (1 d)	Air	4,460	6.54 ± 0.8	2.68 (2.3–3.5)
	10% CO_2	4,622	11.33 ± 1.4	2.32 (2.1–2.7)
Egg (2 d)	Air	2,977	8.05 ± 1.4	2.77 (2.3–4.2)
	10% CO_2	2,917	16.3 ± 1.7	1.63 (1.6–1.7)
Egg (4 d)	Air	3,699	12.09 ± 2.0	2.87 (2.4–4.4)
	10% CO_2	4,089	10.0 ± 1.8	2.61 (2.2–3.9)
Third instar	Air	798	7.09 ± 1.1	0.66 (0.6–0.9)
	10% CO_2	841	7.28 ± 1.7	0.77 (0.6–1.0)
Fifth instar	Air	800	3.9 ± 0.3	1.68 (1.2–2.2)
	10% CO_2	840	5.5 ± 0.4	1.36 (1.2–1.6)
1-d-old pupae	Air	1,097	3.57 ± 0.4	4.01 (3.1–6.02)
	10% CO_2	1,113	2.04 ± 0.4	4.79 (2.9–18.2)
3-d-old pupae	Air	672	3.96 ± 0.6	3.63 (2.9–5.1)
	10% CO_2	1,518	2.32 ± 0.2	7.51 (5.9–10.8)
5-d-old pupae	Air	954	5.81 ± 0.6	1.31 (1.2–1.5)
	10% CO_2	1,094	1.98 ± 0.2	3.89 (3.1–5.4)
Adult	Air	1,154	8.95 ± 1.7	0.34 (0.3–0.6)
	10% CO_2	1,000	9.75 ± 1.1	0.17 (0.16–1.9)

[a] Total number treated over three replications.

control) and exposed to 5.0% EF at 24°C for 1 or 2 h in glass jars [983 g in 9.45-liter jars for 10% load factor (wt:vol)] as described previously for target pests with three replications. Empty treatment jars and jars containing grapes with a 10% load factor were checked for sorption of EF. After treatment, grapes were vented for 1 h at 24°C and then placed in commercial grape bags (vented) and boxes and stored for 2 or 28 d at 2°C before quality evaluations to simulate air or sea shipment, respectively. Quality evaluations also were made after an additional 2 d at 20°C for both storage time points to simulate a marketing period. Thompson Seedless grapes were evaluated for rachis browning, stem browning, bleaching, berry shatter, and decay using a subjective scale: none (1), slight (2), moderate (3), or severe (4). Berry shatter was measured by gently shaking intact clusters over an empty tray for 5 s and then rating the degree of shatter as none (1), slight (2), moderate (3), or severe (4). Firmness was measured subjectively by squeezing the berry between the thumb and index finger and scored as firm (1), slightly soft (2), or soft (3).

Data were analyzed by JMP Statistical Discovery by using analysis of variance (SAS Institute 1995). A Tukey–Kramer significant difference test (honestly significant difference) was used for means separation of fruit quality data.

Results

Western flower thrips eggs were the most tolerant life stage to EF fumigation, and adults were the least tolerant life stage (Table 1; Fig. 1). For Pacific spider mite, the eggs and juvenile stages were the least tolerant life stages, and the adults were the most tolerant. Pacific spider mite protonymphs and deutonymphs were intermediate and similar in their tolerance to EF fumigation. For grape mealybug, the eggs were by far the most tolerant, and the crawlers were the least tolerant (Table 1; Fig. 1). Omnivorous leafrollers were generally more tolerant than other species tested; therefore, these were exposed to EF for 2 h compared with 1 h for other species. The 1- and 3-d-old pupae were the most tolerant followed by the eggs and then the 5-d-old pupae (Table 2; Fig. 2). There was little difference in tolerance between 1-, 2- and 4-d-old omnivorous leafroller eggs. The adult omnivorous leafrollers were by far the least tolerant life stage.

The addition of 10% CO_2 to EF during fumigation had varying affects on the efficacy of the fumigation depending on the pest species and life stage. The addition of CO_2 significantly increased the efficacy of EF on western flower thrips (prepupae and adults), grape mealybug (crawlers and adults) (Table 1; Fig. 1), and omnivorous leafroller 2-d-old eggs as judged by nonoverlap of 95% CI for LC_{99} (Table 2). However, the addition of CO_2 decreased the efficacy of EF for 3- and 5-d-old omnivorous leafroller pupae, some of the most tolerant species and life stages (Table 2; Fig. 2). For the remaining species and life stages, the addition of CO_2 had no significant effect.

There was no significant difference in berry browning, shatter, firmness, bleaching, or decay in 'Thompson Seedless' grapes for any treatment or storage time, although there was a trend toward higher levels of berry browning, shatter, and decay and lower firmness in grapes treated with EF for 2 h (Table 3). Exposure to 5.0% EF resulted in increased browning of the rachis and stems at the first evaluation (2 d after treatment) (Table 3). Any further storage of the grapes resulted in increased rachis browning in control fruit, similar to levels in treated fruit. Ethyl formate concentration in treatment jars peaked between 20 and 40 min. The concentration remained steady in empty jars and decreased 23% in treatment jars containing grapes (Fig. 3).

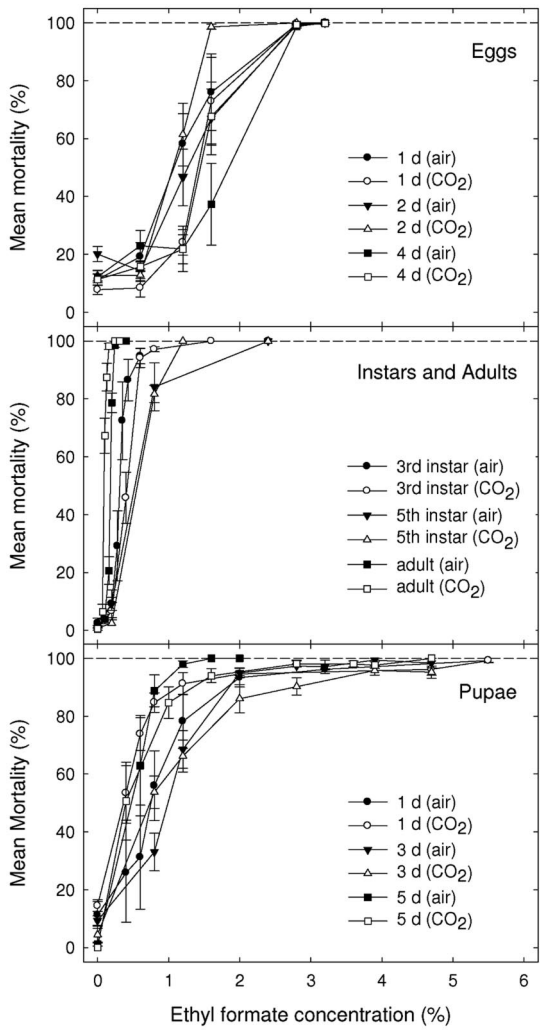

Fig. 2. Mortality of various life stages of omnivorous leafroller exposed to a range of EF concentrations for 2 h at 24°C in air or in 10% CO_2 balance air.

Discussion

Our results indicate that response by pest organisms to EF exposure is variable between species as well as life stages within species. This concurs with the findings of Vincent and Lindgren (1972). In our study, grape mealybug crawlers were the most susceptible to EF exposure, followed by western flower thrips adults and second instars. Aharoni et al. (1980), Stewart and Aharoni (1983), and Stewart and Mon (1984) reported similar results with western flower thrips and green peach aphid, although their fumigations were under vacuum. Omnivorous leafroller life stages were more tolerant to EF treatment and thus were exposed for 2 h compared with 1 h for other species.

Western flower thrips (prepupae and adults), grape mealybug (crawlers and adults), and 2-d-old omnivorous leafroller eggs had significantly lower LC_{99} values when 10% CO_2 was added with EF than with EF in air. However, 3- and 5-d-old omnivorous leafroller pupae, among the most difficult to kill with EF, had higher LC_{99} values with EF in 10% CO_2 than EF in air. Other species and life stages tested were unaffected by the presence of CO_2.

Elevated CO_2 has been shown to improve the efficacy of some fumigants, such as methyl bromide, acetaldehyde, phosphine, and ethyl formate (Athie et al. 1998, Rajendran and Muthu 1989, Scheffrahn et al. 1995). However, other evidence indicates that for some species or life stages within species, combining increased levels of CO_2 with fumigants has little or no effect or may decrease efficacy (Jones 1938, Soderstrom et al. 1991, Simpson et al. 2003). Target pest response to specific concentrations of CO_2 combined with EF has been shown to vary between pests (Simpson et al., 2004). This information is especially important for fumigants such as EF that require increased concentrations of CO_2 to be present in the commercial formulation to decrease flammability. Additional information on the response of economically important pests to EF with various concentrations of CO_2 will assist in the development of such commercial formulations.

Our results with table grapes indicate that exposure to EF at concentrations as high as 5.0% for 1 or 2 h are well tolerated with the exception of increased rachis browning. Rachis browning was significantly higher in EF treated fruit 2 d after treatment; however, after further storage, the rachis of untreated fruit was similarly browned. The LC_{99} for all target pests and life stages fell below 5.0% EF, with the exception of omnivorous leafroller pupae, especially when treated with EF and 10% CO_2. Tolerance to EF varies with commodity. Strawberries showed little to no calyx damage and no berry damage when exposed to three 1-h applications of 0.8% EF at 24°C (Simpson et al. 2004). Walnuts, almonds, and pistachios showed no negative effects from multiple applications of EF (K. Damcevski, personal communication). Vacuum fumigation of packaged lettuce with 0.5% EF for 1 or 2 h to control green peach aphid did not result in a detectable loss of quality in the commodity and was effective in controlling the pests (93–98% mortality) (Stewart and Aharoni 1983, Stewart and Mon 1984).

California table grapes are sometimes exposed to 6% CO_2 with 1% SO_2 for spider control before export. The addition of this pretreatment before the EF treatment may increase mortality and reduce the concentration of EF needed for control. Future studies are planned to determine the effects of a pretreatment of 6% CO_2 with 1% SO_2 before exposure to EF for table grape pest control. However, ethyl formate is a potential fumigant for a number of crops, most of which do not tolerate SO_2 fumigation. Tolerance of these other crops to EF fumigation has yet to be determined. Ethyl formate has been registered in Australia and New Zealand for control of postharvest pests as Vapormate (BOC Ltd., Wagga, Australia), containing 16.7% EF by weight dissolved in liquid CO_2

Table 3. Rachis browning, berry browning, shatter, firmness, decay, and bleaching of Thompson Seedless grapes exposed to 5.0% EF for 1 or 2 h at 20°C

EF concn (%)	Treatment		Storage		
	Exposure time (h)	2 d at 2°C	2 d at 2°C + 2 d at 20°C	28 d at 2°C	28 d at 2°C + 2 d at 20°C
Rachis browning[a]					
0.0		1.5b	2.2b	2.5a	2.2a
5.0	1	3.2a	3.2ab	3.2a	2.7a
5.0	2	3.4a	3.7a	3.6a	3.3a
Berry browning[a]					
0.0		1.0a	1.3a	1.3a	1.7a
5.0	1	1.0a	1.0a	1.3a	1.0a
5.0	2	1.4a	2.0a	2.2a	1.8a
Shatter[b]					
0.0		1.4a	1.0a	1.6a	1.5a
5.0	1	1.7a	1.5a	1.7a	2.1a
5.0	2	1.2a	1.1a	2.2a	2.3a
Firmness[b]					
0.0		1.0a	1.2a	1.2a	1.7a
5.0	1	1.0a	1.0a	1.4a	1.3a
5.0	2	1.0a	1.2a	2.1a	1.9a
Decay[a]					
0.0		1.0a	1.0a	2.0a	2.7a
5.0	1	1.0a	1.3a	1.3a	1.5a
5.0	2	1.0a	2.0a	2.7a	2.6a
Bleaching[a]					
0.0		1.2a	1.0a	1.0a	1.0a
5.0	1	1.0a	1.0a	1.0a	1.0a
5.0	2	1.0a	1.0a	1.0a	1.0a

Storage after treatment as indicated. Means in a column (three replications) followed by the same letter are not significantly different at the 5% level.

[a] Damage/shatter score: 1, none; 2, slight; 3, moderate; and 4, severe.

[b] Firmness rating: 1, firm; 2, slightly soft; and 3, soft.

solvent/propellant (Ryan and Bishop 2003) and registration in the United States is being pursued by BOC Ltd. (Chatswood, New South Wales, Australia). If registration in the United States is successful, current fumigation chambers and equipment used in California could potentially be modified for use with Vapormate.

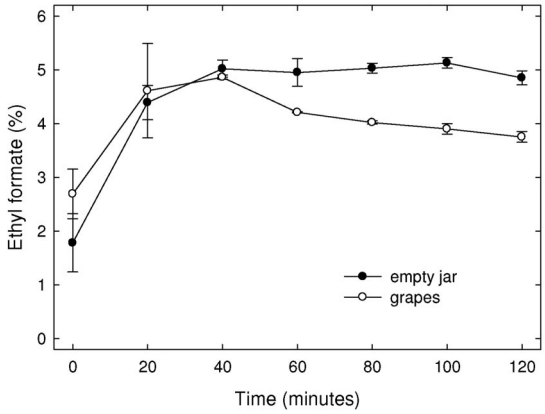

Fig. 3. Headspace EF concentration (%) over time (2 h) in an empty treatment jar or a treatment jar containing table grapes [10% load factor (wt:vol)].

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